# A FLUIDIZED POWDER APPARATUS FOR DISTILLING SAWDUST



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TABLE OF CONTENTS

# LIST OF FIGURES

# A Fluidized Powder Apparatus for Distilling Sawdust

## Chapter I

## Introduction

The lumber industry is one of the principal industries of the United States, and it is of particularly great importance in certain regions of the country. The South is one of these regions; consequently, its economic well-being is affected by that of its lumber industry. Any means by which the economic position of the industry can be improved, therefore, will be of general benefit to the people of the South.

Perfecting a profitable use for a wasted by-product of an industry is an important means of bettering its economic position. A lumber industry by-product which is now largely wasted is the sawdust and other small material produced in sawing, planing, and similar operations. It is customary in many sections of the country to convey the sawdust pneumatically from the scene of the operations to a dumping area, where it accumulates in large piles. Occasionally, the sawdust is burned to produce useful heat, but ordinarily it is left in the piles to decay, constituting a constant fire hazard. Several attempts have been made to utilize this waste sawdust, such as pressing it into

building blocks or fuel briquettes. These attempts have been successful to varying degrees, depending on local economic conditions.

It has been suggested that the waste sawdust might be subjected to destructive distillation. Currently used methods of wood destructive distillation are not applicable, principally because of the poor heat transfer characteristics of a closely packed agglomeration of sawdust.

The inherent excellent heat transfer characteristics of the fluidized solids technique, as it has been applied to fluid catalytic cracking of petroleum oils, suggest a means of overcoming this poor heat transfer and accomplishing the desired destructive distillation. The simplified flow sheet of a full scale plant for fluidization sawdust distillation might be visualized as something on the order of that shown in Figure 1. Such a plant might operate something along these lines. The sawdust feed, sized properly, enters down the standpipe (1), Figure 1. The standpipe would be of sufficient height to give a proper static head, and its flow would be regulated by the slide valve indicated, SV. Air would enter the system through the blower and pick up a regulated amount of charcoal from standpipe (2). The air-charcoal mixture is brought to oxidation temperatures in the furnace, and the charcoal is practically completely oxidized. The charcoal-air mixture would be so regulated as to achieve complete consumption of

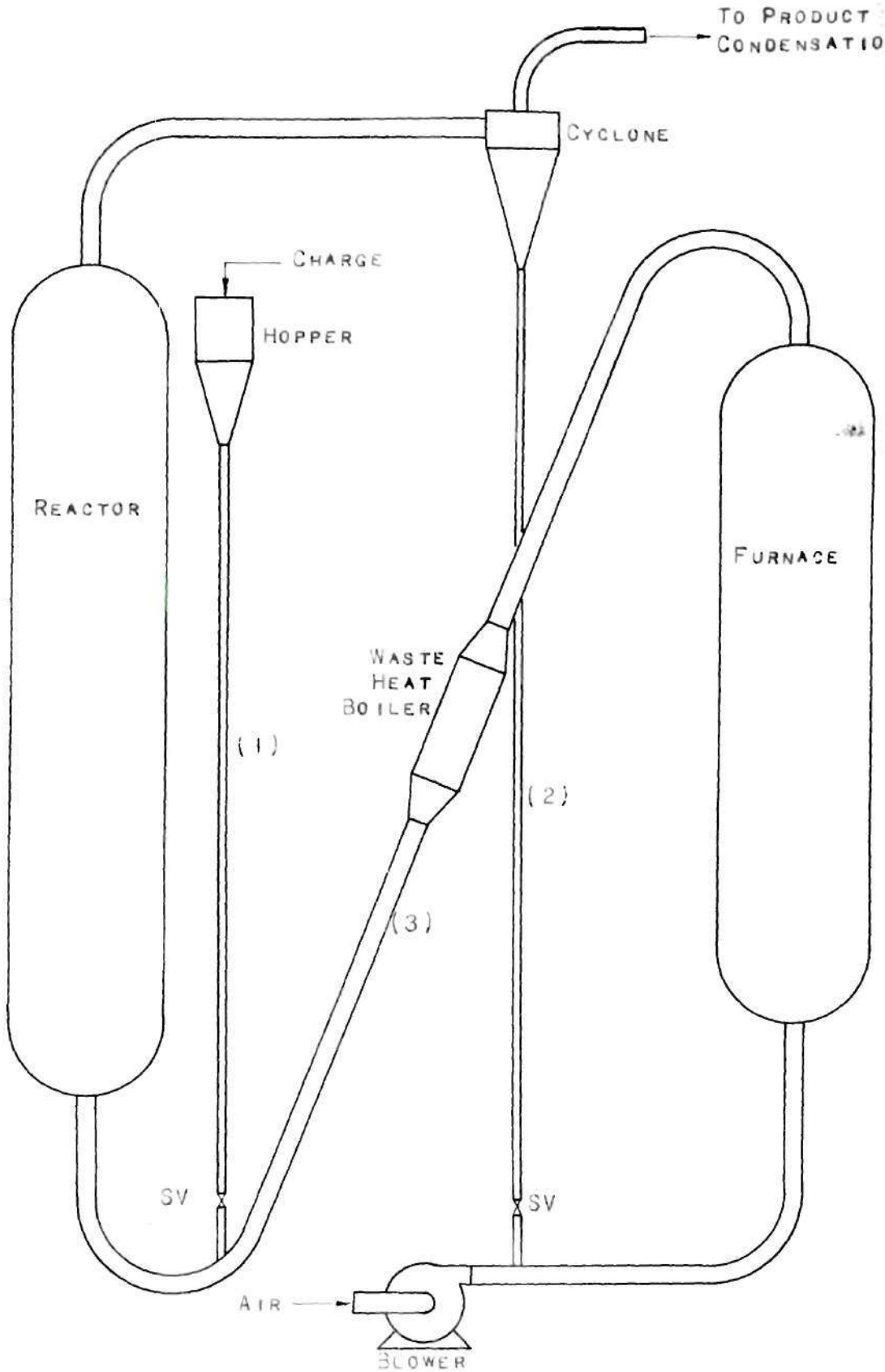



FIGURE 1
DIAGRAM OF A FULL SCALE PLANT

the oxygen present. The flue gases from the furnace pass overhead through line (3) and into the waste heat boiler. Here, heat would be removed until the gases were at a desired temperature. From the boiler the gases would pass to the foot of the standpipe (1), where they would pick up the regulated flow of feed. The gas-feed mixture would be carried over to the reactor where conditions would be regulated for carrying out the distillation. Products from the reactor would pass overhead to a cyclone separator where the charcoal residue from the distillation would be separated and the gaseous products would be sent to condensation and fractionation.

The mechanics of the flows of the gases and solids in the plant would be similar to those in the fluid catalytic cracking process for hydrocarbons, as described by Murphree et al (11). The fluidized solid technique offers several advantages, prominent among which are excellent heat transfer, extreme flexibility, and accurate control of reaction time, rate, and temperature. This accurate control and the large ratio of reaction surface to weight of reactants should produce yields of better quality and quantity than obtainable by conventional wood distillation.

The economic phases of this process are very complex and will probably require as much study, after process design data are obtained, as was devoted to obtaining the design data. The manufacture of synthetic methyl alcohol and

acetic acid is so much more economical than obtaining them from wood by present methods of distillation, that, at the present time, practically the only wood distillation plants being operated are those whose main product is charcoal. Improvement of wood distillation processes, however, might change the situation. There is, for instance, a new furnace process under consideration, which gives better operation and yields and promises to compete with the synthetics (1). The fluidized sawdust process should give even more economic operation, especially considering the low price at which the waste sawdust should be obtainable. Other main economic factors which must be considered include the market value of the charcoal produced as compared with its value as fuel, the location and accessibility of raw materials and markets, and the competition from new synthetic processes, such as the Hydrocol process which will produce a number of alcohols, aldehydes, and organic acids as by-products.

Since the proposed process of destructive distillation of fluidized sawdust seems to offer favorable prospects, it was decided that a more comprehensive investigation should be initiated, in order to obtain data which would offer a suitable foundation for process design purposes. Pursuant of this purpose it is necessary to determine the effect of a number of process variables, such as temperature, time, sawdust particle size, and type of wood charge. Since these determinations are best made experimentally, a suitable apparatus is necessary.

The purpose of this paper is to describe the design, construction, and preliminary operation of such an apparatus.

As shown in Figure 1, the full scale plant would include units for both the production of fluidizing gases by combustion of the charcoal produced and the actual destructive distillation process. The experimental apparatus consists only of the destructive distillation phase of the complete plant, so it will not be subject to the severe high temperature and oxidation conditions of the combustion unit.

A review of pertinent literature was not very fruitful. Information on fluidized solids techniques was restricted during the war, and by the time this literature search was made, very little information had been released. The subject is also so new that little information dealing with laboratory scale equipment had been published. Published data on wood distillation were necessarily subject to question in applying them to the problem at hand, because of their being based on a different type of process.

Since the literature yielded very little useful information about those operating variables which had to be known for designing the desired apparatus, it was necessary first to make some preliminary experiments that would yield sufficient data.

Several glass apparatuses were built to study the characteristics of fluidized sawdust under various conditions. Using the data obtained from these preliminary experiments,

a laboratory apparatus for studying the destructive distillation of fluidized sawdust was designed. The apparatus was built and operated on several exploratory runs.

# Chapter II

## Design

At the time when design work on this project was undertaken, the literature contained practically no information useful in establishing a basis for laboratory equipment design. Practically the only pertinent material available consisted of limited construction and operation details of full scale fluid catalytic crackers and occasional vague mention of technical data concerning such units. Thus the literature did not furnish such information as details of laboratory work, relationships between material size or density and gas velocities, equipment sizes and shapes, or particle size relationships. In order to obtain such information, to become familiar with fluidized solids phenomena, and to gain experience with fluidized powdered sawdust in particular, several preliminary apparatuses were built and operated for brief periods of time.

The simplest of these preliminary units is illustrated in Figure 2. The auxiliaries to this unit consisted of a source of compressed air and a regulating valve in series with an orifice meter. These three facilities served to introduce a measured flow of air into the bottom of the experimental unit itself. This unit was constructed of glass and consisted essentially of glass tube some two or three feet long, fitted

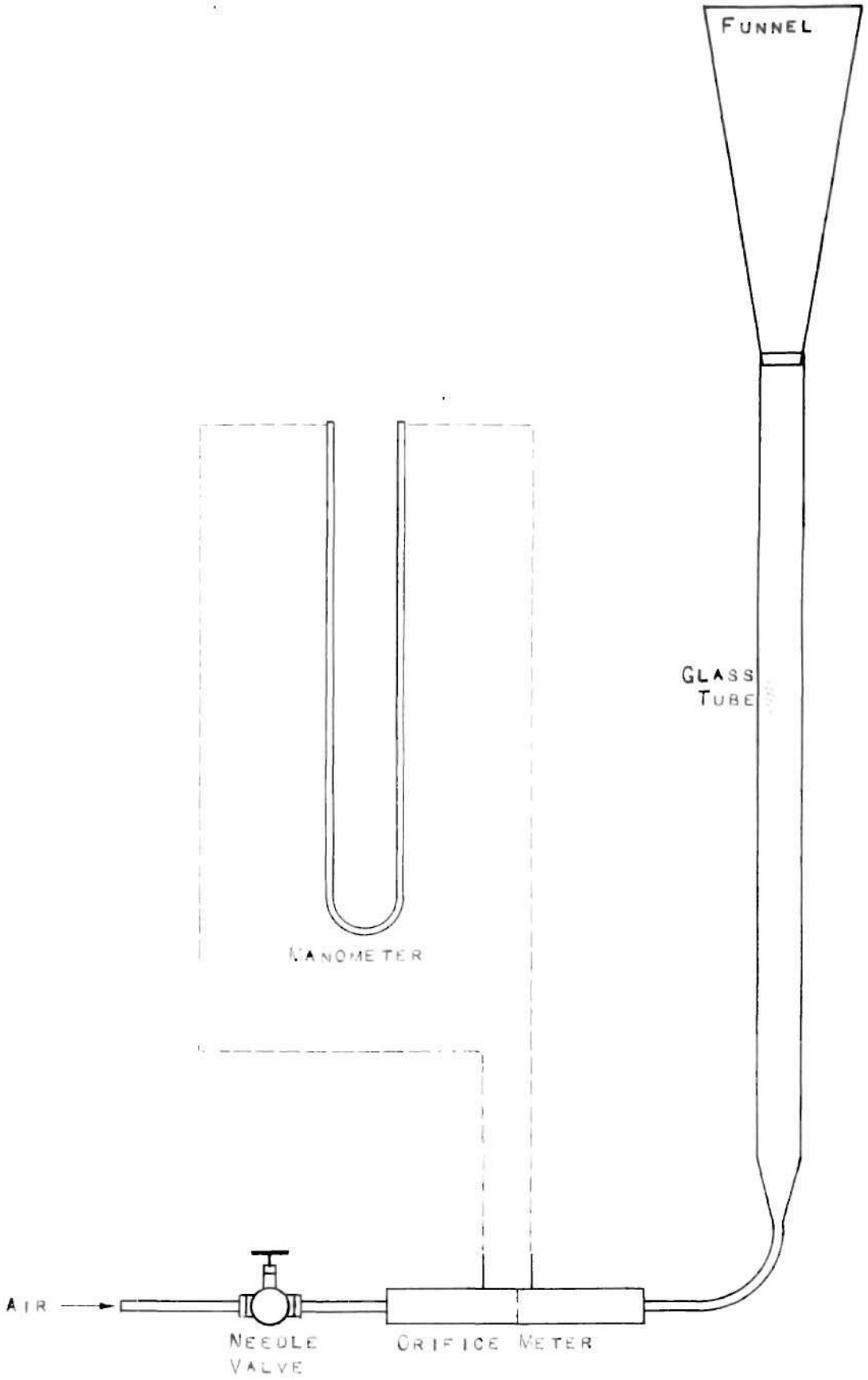



FIGURE 2
FIRST EXPERIMENTAL APPARATUS FOR DESIGN STUDIES

at its bottom end with a reducer for the purpose of enlarging the cross-sectional area of the stream of gas gradually. Tubes of several different diameters were used. The tops of the glass tubes were each fitted with a large metal funnel, wherein the velocity of the gas steam would be reduced and all but the smallest particles of solids would settle out and eventually fall back into the tube.

Operation of this type of apparatus was especially useful in obtaining data on various gas velocities through a bed of powdered sawdust, in observing the action of the bed at various gas velocities, and in determining the effects of various sizes of tubes. As a result of several runs on this apparatus a good idea of the desired velocities for good fluidization was obtained.

The first unit of this type made had a tube 19 mm inside diameter. While it was possible at times to obtain a fluidized bed in this tube, a good deal of trouble was experienced with the solid material forming slugs which broke up the fluidized bed in alternate solid and gas layers. By increasing the internal diameter of the tube to two inches or more, it was found that the slugging trouble was practically eliminated.

One of the apparatuses of this type did not have a gradually changing cross-section in that part of the reactor where the air line was fitted to the tube. It was found that this produced an unsatisfactory type of channeling action in

the bottom of the tube up to that point at which the gas had had a chance to adjust itself to the enlarged cross-section of the tube.

Observation of the fluidized bed action during these experiments showed that a velocity which is great enough to produce a well fluidized bed is also so great as to cause an appreciable quantity of solids to be carried up out of the bed. It was obvious from this observation that operating with satisfactory fluidization would require facilities for removing solids carried out of the bed and returning them to the bed.

As a further extension of this type of experimental apparatus, it was decided to make a study of the action of powdered sawdust in a similar apparatus, but one which could be heated. Some idea of the feasibility of the proposed process might be obtained thereby, as well as useful design data. The set-up of this equipment is shown in Figure 3. In order to supply a non-oxidizing atmosphere in the tube, nitrogen from a high pressure cylinder was used as the fluidizing agent. The reactor tube itself was much like that in Figure 2 used in preceding experiments, except that it was wound with an electric heating coil, and it had a chromel-alumel thermocouple placed inside with its juction just about the midpoint of the reactor. The nitrogen flow was controlled by the reducing valve on the cylinder and measured by an orifice meter. Several runs were made using sawdust of 42-100

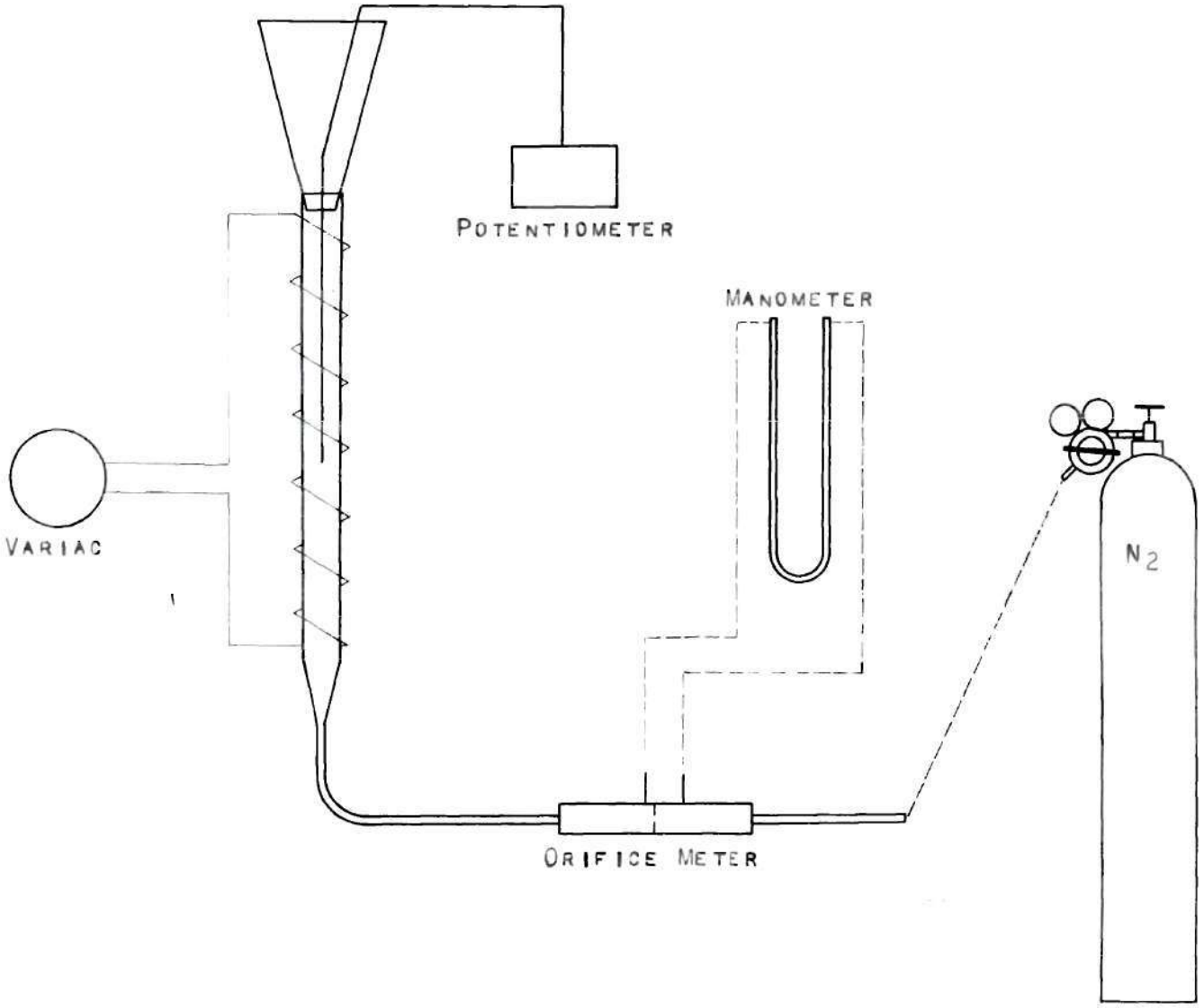



FIGURE 3
HEATED EXPERIMENTAL APPARATUS FOR DESIGN STUDIES

mesh and temperature around 300°C.

Although operation of this apparatus was never as successful as might be desired, some very encouraging results were obtained. A voluminous production of vapors during one run established quite satisfactorily that destructive distillation was taking place. Runs on this apparatus also confirmed the temperature range at which it was expected that destructive distillation would take place. These two observations, coupled with the generally encouraging operation of the apparatus, seemed to justify further work on the problem.

Concurrent with the above mentioned work, experiments were being carried out in cooperation with Mr. E. M. Koeritz in an effort to determine a practicable laboratory scale apparatus for fluidizing powdered sawdust. The course followed in this work was tantamount to scaling down full scale operation, as practiced in the early fluid catalytic cracking units. Several models were built during the course of this experimental work, but all were similar to the final apparatus which evolved from this work and is shown in Figure 4.

Except for the control valves, the cyclone, and the orifice meter, this apparatus was constructed entirely of glass. The column (1) was used for holding the sawdust collected in the cyclone. This sawdust was allowed to fall through the plug valve, being controlled by this valve, and

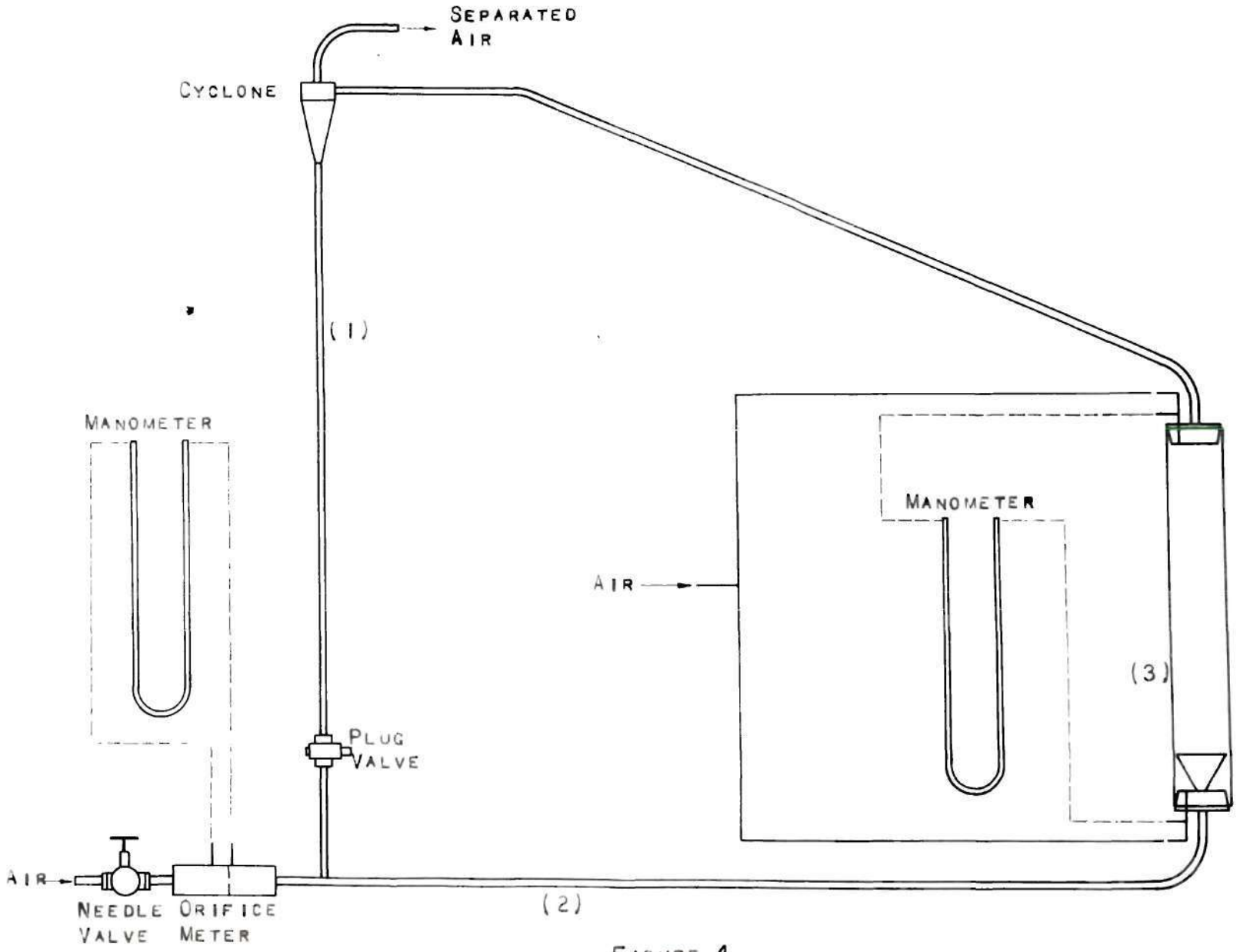



FIGURE 4
THIRD EXPERIMENTAL APPARATUS FOR DESIGN STUDIES

was picked up by the steam of air flowing horizontally along (2). The air flow was controlled and measured by the facilities provided. It carried the sawdust which it had picked up into the fluidized bed (3). The reduced velocities in the enlarged section (3) maintained therein a fluidized bed of sawdust. As the sawdust was carried out of the fluidized bed, it traveled up through the top line to the cyclone, where the air was separated and the sawdust was regulated into (1). It was interesting to note that while the fluidized bed was in operation, a good amount of the sawdust collected in the annular space between the distributing funnel and the wall of the cylinder in the bottom of (3). The fluidized bed column (3) was equipped with pressure taps at both ends to measure the pressure drop across the fluidized bed. Each tap had a blow-out attachment for keeping it free of sawdust by admitting a steady, regulated flow of air.

The sawdust used in these runs contained particles which varied considerably in size, perhaps over a range from 10 to 100 mesh. Some satisfactory operation was obtained with this apparatus, and it was possible for Mr. Koeritz to use it for some related experimentation; however, the experience gained trying to operate it satisfactorily indicated that this approach to a laboratory apparatus was not workable. In order to gain the static heads necessary for the flow in the apparatus of Figure 4, it was necessary to make the standpipes quite tall. For a laboratory apparatus to have so much

external piping would require excess heights, and the large amount of small diameter piping would produce extremely excessive heat loss with the attendant condensation of liquid product in undesirable portions of the apparatus. Also, this type of apparatus is for cyclic operation in which the charcoal produced would be burned to produce fluidizing gases. For the laboratory apparatus it was not considered desirable to undertake cyclic operation. An apparatus which consisted of mainly the fluidized reaction bed and which would have a minimum amount of external piping seemed to be more in line with what would be needed.

After the completion of these preliminary experimentations, the design of the final apparatus was undertaken. In addition to being capable of reasonably exact duplication of the process under consideration, prime requisites of an apparatus for obtaining good experimental data are flexibility of operation and the best possible control. Retaining these ideas as guides, several basic requirements for the apparatus were established.

It had been observed that a reactor diameter of about two inches seemed to be about the minimum for good fluidization. Since it was desirable to minimize heat and fluidization medium requirements by keeping the reactor diameter as small as possible, it was decided that the inside diameter of the reactor should be about the minimum two inches.

The return to the fluidized bed of particles which

would necessarily leave it at fluidization gas velocities could be accomplished in several ways. It was considered best, however, to effect this return in a manner which would necessitate no complicated equipment and which would return the greatest part of the entrained material directly to the reactor by a simple means. A tapered expansion of the reactor to a section of greater diameter, wherein the reduced velocity of the fluidizing gas would drop out most of the entrained material, was chosen as being best. Experience from the preliminary experiments indicated that a total angle of 20° in the tapered interconnecting section offered an approximate optimum between the length of the tapered section and the desire to make this angle as small as possible. A minimum angle is desirable in order to promote the flow, by gravity, of the recovered particles to the fluidized bed.

Since it was obvious that some very small particles would be carried through the enlarged disengaging section above the reactor, the addition of a small cyclone separator following the disengaging section was deemed necessary. The small size of these particles necessitated a small cyclone having a diameter of about three inches. It was estimated that particles which could pass through such a small separator would not cause any trouble in the apparatus at a later point.

In line with the idea of making the apparatus flexible in operation, it was desirable to design it so that it could be run for either continuous or batch operation. Operating in these two manners with one apparatus presented two requirements which had to be met. First, for continuous operation, it had to be possible to charge fresh material and remove reacted product, while the unit was operating. Secondly, for batch operation, there had to be no place within the apparatus where fresh charge could collect in appreciable quantities and not be reacted. The first of these two requirements was met by means of a double valve and hopper arrangement for feeding the apparatus and removing reacted sawdust, the second, by keeping at a minimum the volume of the reactor which was not directly in the path of the fluidizing gases.

Reaction temperatures in the apparatus were to be relatively high, in the neighborhood of 300°C. For this reason and in order to obtain good temperature control, it was decided to heat the reactor with an external electric resistance type heater. In order to enhance further the inherent good controllability of such heaters, the reactor was actually wound with separate heaters, one wound on top of the other and separated by asbestos insulation. Each heater was connected to an auto-transformer of the Variac type, in order to procure further controllability.

In an effort to prevent the undesirable condensation of volatile reaction products in the apparatus between the

reactor and the condenser, it was decided that a third electric heater should be used. It was wound around the disengaging section, the cyclone separator, and their interconnecting piping. The third, or auxiliary heater, was also connected to a Variac type autotransformer.

During preliminary experiments using plug valves for controlling the flow of sawdust through round conduits, it was found that the rectangular shape of the bore in the usual plug caused the solid material to pile up and plug the line. In order to avoid this trouble in the experimental apparatus, plug valves having full area round bores through their plugs were specified.

During continuous operation it is necessary to admit fresh sawdust to the apparatus and to withdraw reacted product in regulated amounts as steadily as possible. This can be accomplished by changing the cross-sectional area of the conduit which is carrying the solid material. The means devised for doing this was a slide valve which consisted mainly of a thin metal plate capable of blocking from 0 to 100% of the area of the conduit, by sliding in a plane perpendicular to the axis of the conduit. This slide valve had to be constructed so that there was little possibility of any sawdust getting into tight places and jamming its operation. A valve of this type was designed.

An important part of the data to be obtained from the apparatus was information on the various operating conditions.

The following data were selected as being pertinent and means for obtaining them were included in the design: temperatures at various points in the reactor, in the disengaging sections, and in the liquid products condenser outlet; the pressure drop across the reactor; the flow of gas into the reactor; and the amount of residue gas leaving the condenser.

As noted in the literature (11) and during operation of the apparatus shown in Figure 4, pressure taps which open into a fluidized bed become clogged with particles of the fluidized material; therefore, it is necessary to provide a means of blowing out such taps and keeping them open. The reactor bed pressure drop taps in the apparatus were designed with double connections, one connected to a monometer and the other to a source of high pressure blow-out gas.

The various design considerations discussed in this chapter were all combined with the knowledge gained during the preliminary experiments and a final plan for the apparatus was formulated.

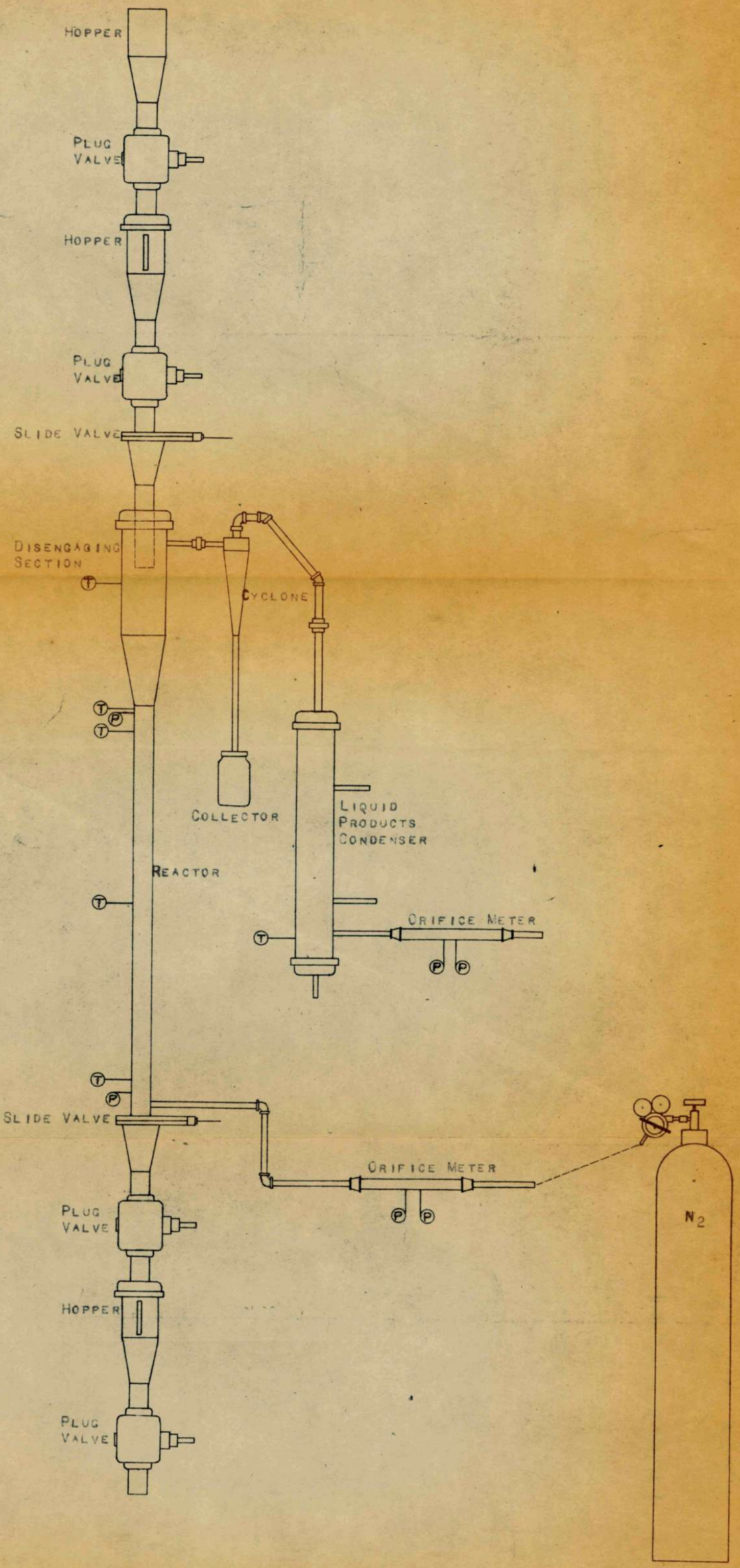
# Chapter III

## Construction

Materials and methods of construction were the primary consideration before starting fabrication. Since the unit was to be only a laboratory model with a relatively short total time of operation, it was considered that mild conditions of corrosion or errosion could be ignored. This consideration dictated the use of the least expensive and most readily available materials, such as black iron and copper. It was not expected that the unit would operate at pressure greatly exceeding atmospheric; therefore, screwed pipe joints would be permissible. Welded joints were used as much as practicable to guard against the possibility of leakage and to facilitate construction operations. It was also necessary to consider the effect of heat on the material of the reactor and other heated parts. Since it was not expected that reaction temperatures would rise above about 300°C, steel pipe was deemed satisfactory for the heated parts of the apparatus.

Figure 5 is a scale drawing illustrating the entirety of the final apparatus itself and most of its auxiliary parts. The heart of the whole unit is the reactor. Fabrication was begun with this section of the equipment. It consists of a four feet long piece of two inch standard pipe, threaded at

the lower end with a standard pipe thread and with a reducer welded to its top end. It was drilled at the points indicated by the "T" and "P" flags, and copper tubing was welded into the holes for thermocouple wells and pressure taps, "T" flags indicating thermocouple wells and "P" flags pressure taps. The former are 1/4 inch diameter, and the latter are 3/16 inch diameter. The disengaging section, composed of a piece of five inch standard pipe about 14 inches long, was welded to the top of that reducer which was welded to the reactor. The disengaging section was also equipped with a welded thermocouple well. The three hoppers were made separately and consisted of six inch sections of four inch standard pipe. A reducer and a nipple were welded to the bottom of each hopper, and the nipple was threaded at its lower end. The two hoppers, which were between two plug valves, were also threaded at their top ends and fitted with pipe caps containing a welded nipple. These two hoppers were also fitted with two plexiglass windows each for observation of their interiors. All of the short pieces of pipe connecting the various hoppers and valves are two inch standard. The plug valves shown are of the round port, full area type, and the slide valves were made according to an original design. The hopper-valve auxiliary combinations were attached to the reactor and disengaging section, all joints being welded except for the screwed pipe caps and the extrances of two inch pipes into the various valves.




SCALE: 1"= 1FT.

FIGURE 5

A FLUIDIZED POWDER APPARATUS FOR DISTILLING SAWDUST

The two inch pipe into the disengaging section was extended as shown in order to prevent short circuiting of the incoming feed out the pipe to the cyclone.

A simple, braced, rectangular, angle iron frame was made and bolted to the floor. Its construction can be seen from consulting the frontispiece or Figure 11. The reactor and its hopper-valve auxiliaries were mounted on this framework by means of two diametrically opposite wing type braces which were welded onto the disengaging section.

The cyclone separator was mounted on the outside of the rectangular frame, being actually hung from its connecting piping and without other support. The cyclone itself was fabricated of light weight sheet metal, welded throughout. It was designed according to more or less standard design of cyclones. A piece of metal tubing was welded to the bottom of the cyclone, and a screw top, gasketed bottle was attached to the opposite end of the tubing. This bottle was designed to be a collector for the material separated in the cyclone. The entrance and exit pipes were welded into the cyclone; the former was 5/8 inch standard pipe and the latter 3/4 inch.

The 3/4 inch line from the top of the cyclone ran over to the top of the liquid products condenser. The condenser was mounted on the side of the framework by means of a metal bracket and a wooden platform. The 5/8 inch pipe from the side of the condenser was for uncondensed gases. It was welded into the condenser at one end, and it supported at the other

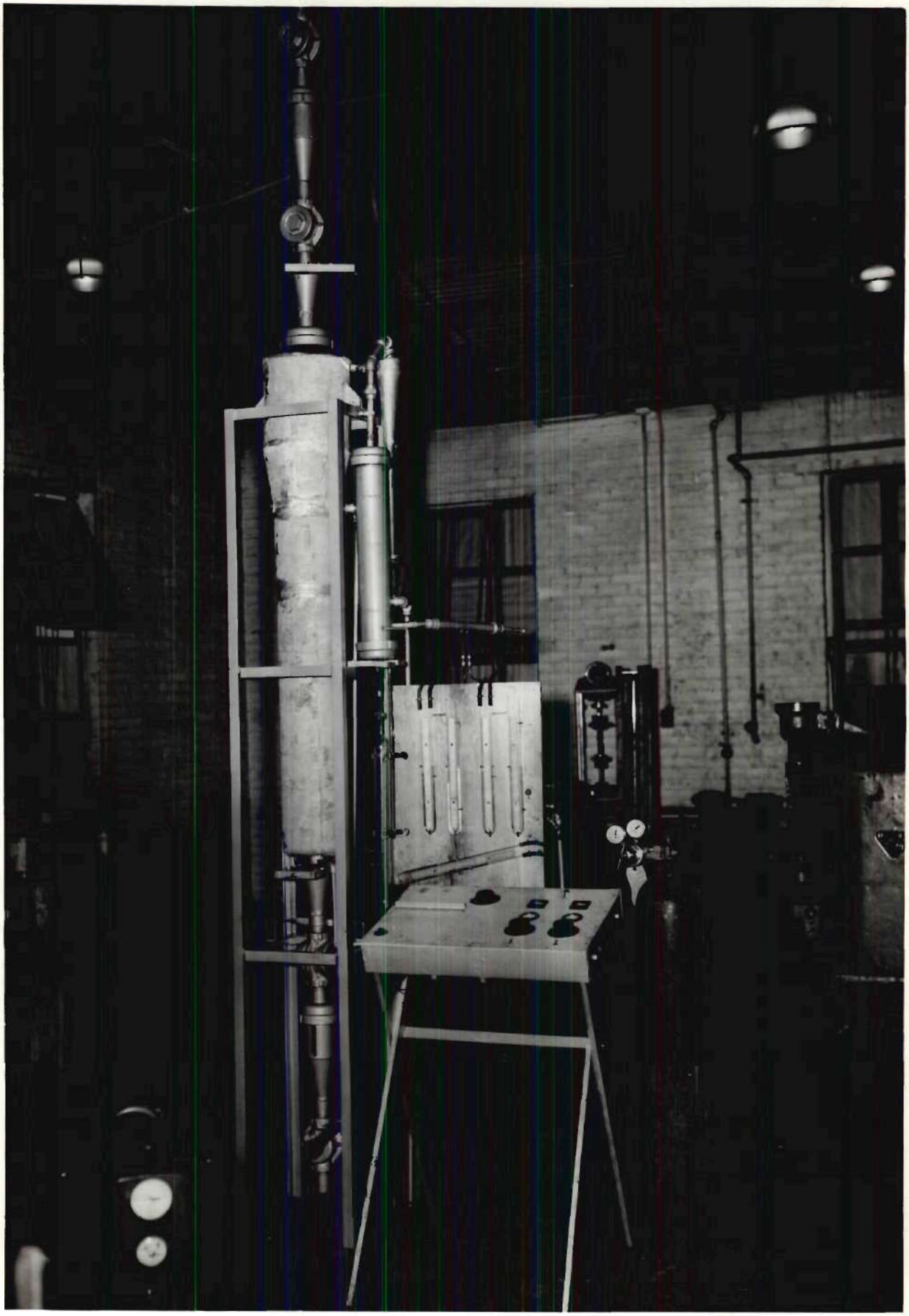
end orifice meter No. 2. The orifice meter itself was made from a piece of one inch pipe about a foot long and threaded at both ends. This piece of pipe was cut in half and an orifice plate was fitted into one of the cut ends. Then the two halves were welded back together, and two copper tubing pressure taps were welded into place on either side of the orifice plate. The orifice meter, No. 1, made to measure the supply of nitrogen to the column, was constructed similarly.

Figure 6 is a diagram of the electrical system of the unit. The reactor, the disengaging section, and the cyclone are sketched in to show the relative position of the wiring. Before wire was wound on any of these three pieces of equipment, they were covered with a coat of Insulite cement. This material was applied to act as a refractory covering and as electrical insulation. A coating of one layer of asbestos paper was applied on top of the Insulite, and the heater wire was wound on top of it. In the case of the reactor where a second heater was wound on top of the first, the two heaters were separated by another layer of asbestos paper. After all the heaters were wound and anchored in place, they were covered with another layer of asbestos paper. In Figure 6, the circuits to the right of the reactor are the power circuits and their auxiliaries, and those to the left are the thermocouples and their auxiliaries. The Variac autotransformers are indicated and the double-pole-double-throw switches of the voltmeter-ammeter circuits of two are shown. The Variac had a 110

volt primary and 0 to 130 volt secondary. The voltmeter and ammeter are appropriately indicated by marking of "V" and "A".

The auxiliaries of the thermocouple circuits were a multiple selector switch and a potentiometer. The thermocouples were chromel-alumel junctions with copper leads from within eight or ten inches of the junction to the potentiometer. The two arms of the junctions themselves were covered with glass capilary tubing of small diameter for the purpose of insulation. They were inserted into the copper tubing thermowells, which had been welded into the equipment so that the junction itself was situated in just about the center of the space in which it was measuring.

Figure 7 shows the details of construction and installation of the expander which is located in the bottom of the reactor. The expander itself is a section of one and a quarter inch pipe, drilled and threaded in the side, and with a round plate welded into its end. As shown in the drawing, the inside above the threaded hole was turned down to a fine edge to aid in expanding the gases. The expander was mounted in the reactor by being placed inside and screwed onto the 3/8 inch pipe which carried in the gases for fluidization. The pipe was then welded into the reactor. The annular space between the outside of the expander and the inside of the reactor and the space under the expander down to the plate of the slide valve offer the only space in the reactor where unreacted sawdust could collect during batch operation.



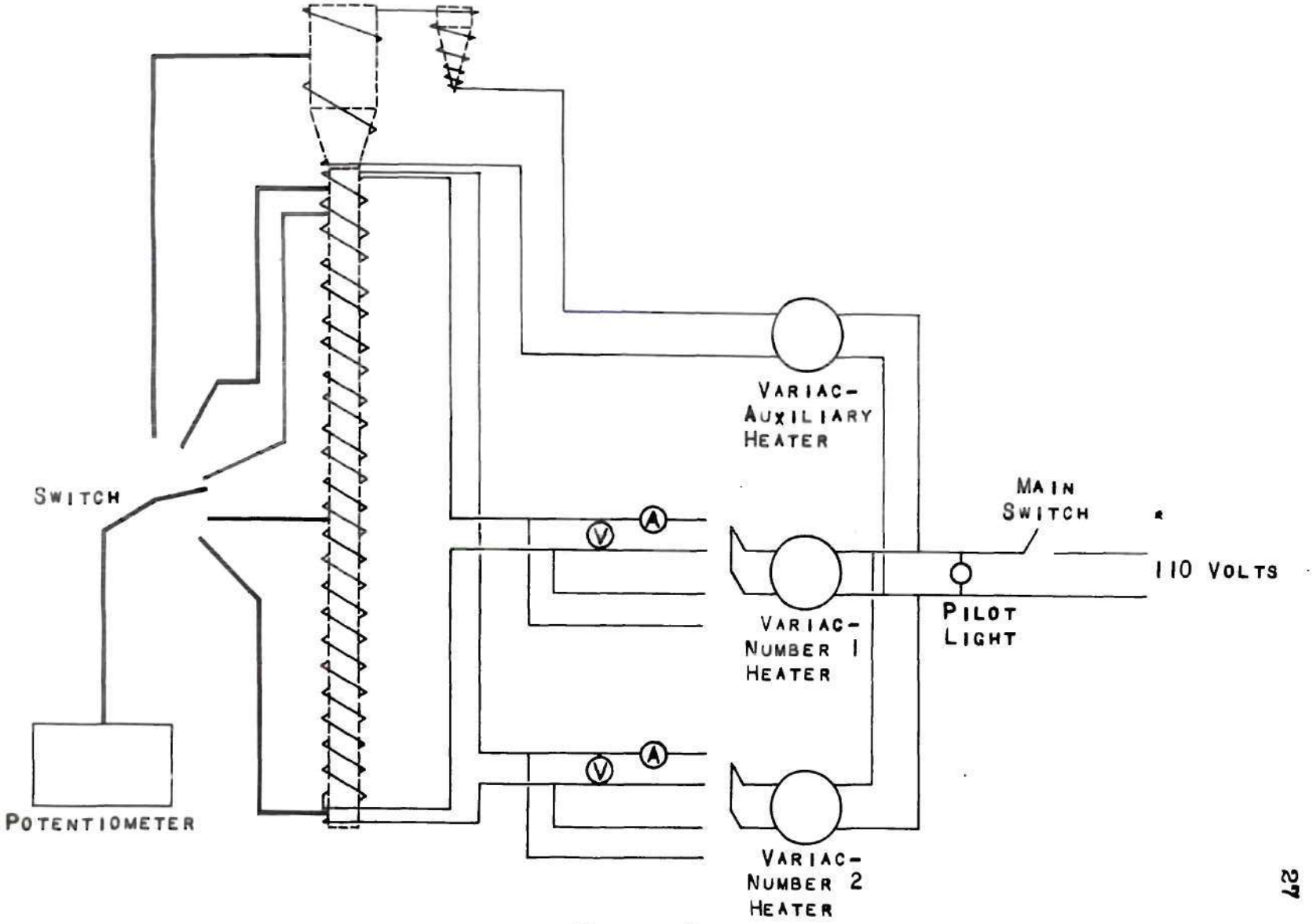
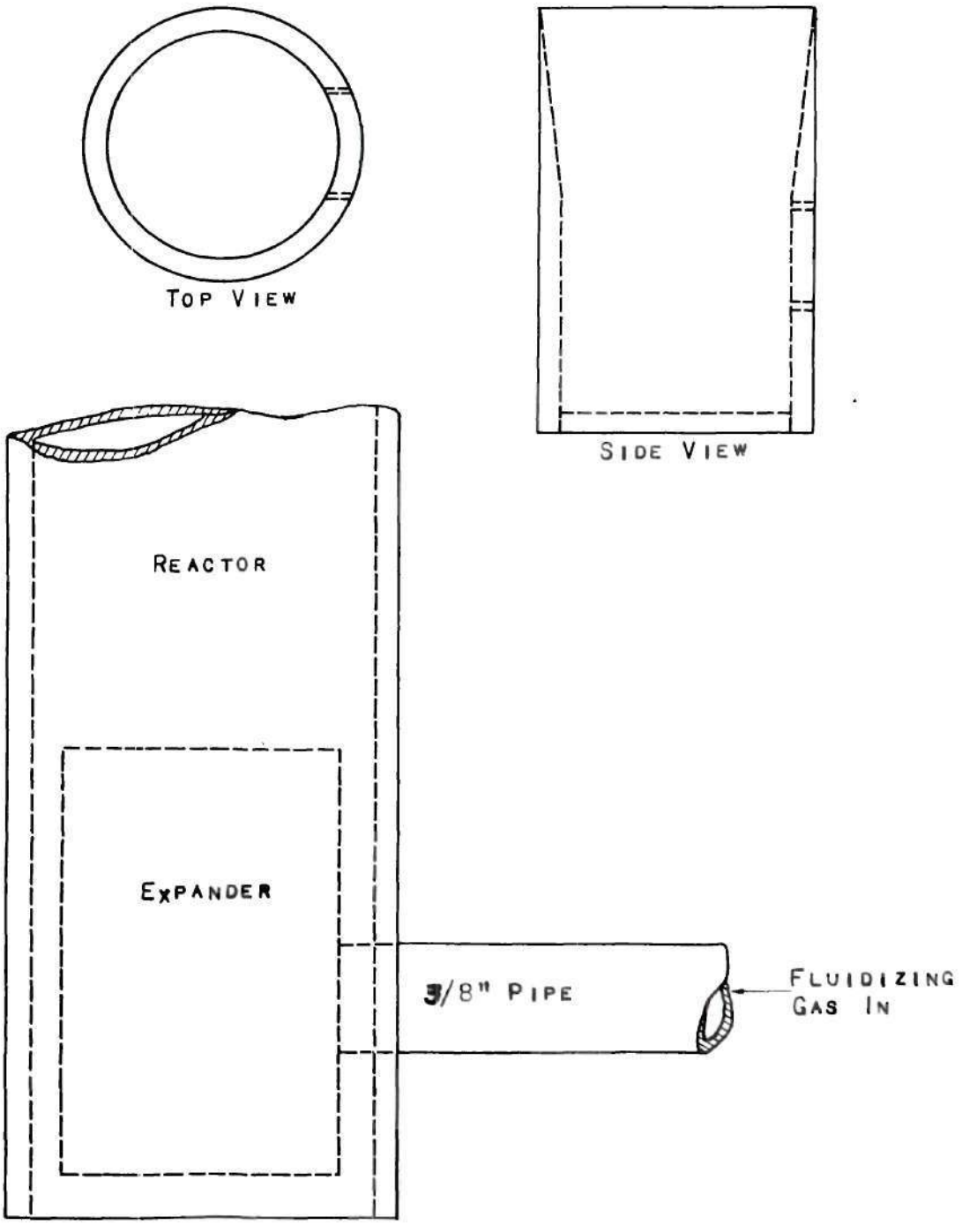



FIGURE 6
ELECTRICAL DIAGRAM OF THE APPARATUS




SCALE: FULL SIZE

FIGURE 7
REACTOR FLUIDIZATION GAS EXPANDER

During continuous operation, these same spaces would act as temporary storage for reacted material which was dropping out from the bottom of the reactor.

Front and side views of the slide valves are illustrated in Figure 8. The body of the valve was made of three pieces of steel. The top half was drilled and tapped for a two inch standard pipe, as shown, and the bottom half was drilled to the inside diameter of a four inch standard pipe. Both halves were drilled and tapped appropriately for the six bolts which hold them together. Each was also milled along one face to form a channel in which the slide might operate. The two halves were bolted together with their milled slots facing so as to form an operating passage for the slide. The two halves were appropriately drilled and tapped on the end through which the slide entered, and the end piece illustrated was mounted with its four screws. The hole drilled through the end piece for the slide handle was enlarged as shown and the first part of it was threaded. This enlarged space was made for placing packing around the slide handle to keep it pressure tight, and a packing gland was made from brass with a hex head at one end and with the other end threaded for entrance into the end piece. The bearing surfaces, where the three sections of the valve body were bolted together, were equipped with asbestos gaskets for pressure tightness and because of the heat which might have an adverse effect on other gasket material.

The slide itself was a piece of brass plate fitted into the two milled slots and drilled and tapped the center of one end to receive its handle. The handle was a piece of small diameter round steel rod. The reducer was welded onto the bottom half of the valve body, in order to reduce the four inch opening therein to two inch standard pipe size. The hole in the bottom half of the valve was made so much bigger than the entrance at the top in an effort to allow free passage of the sawdust and thereby prevent the sawdust from accumulating and blocking the action of the slide.

The two views of Figure 9 illustrate the tube sheet and a cut-away section of the liquid products condenser. The condenser was of course mounted vertically, but it is shown here in the horizontal position for convenience of illustration. The shell of the condenser was made of four inch standard pipe. The two heads were made of steel plate and were both welded into the shell. The nineteen tubes in the condenser were 20 gage copper tubing, 5/8 inch diameter, and 16 inches long. They were welded into both heads. Welding tubes and heads at both ends like this is ordinarily bad practice, but, presumably because of the short length and mild operating temperature of these tubes, there was no trouble during operation. An additional piece of four inch standard pipe, six inches long, was welded onto the ends of the shell in order to provide for distribution of incoming gases and for disengaging space for condensed liquid and cooled gases. The two ends were threaded

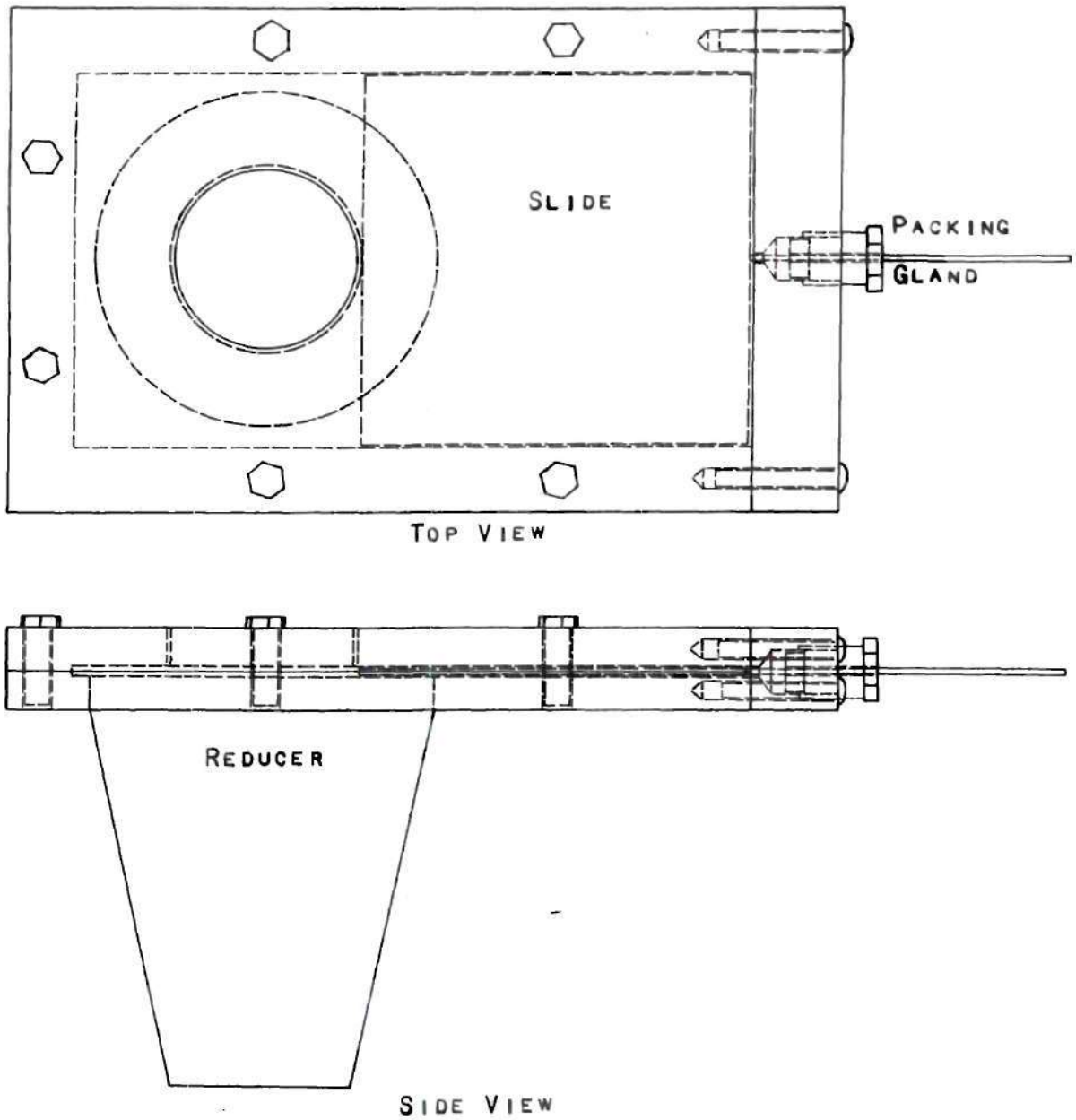
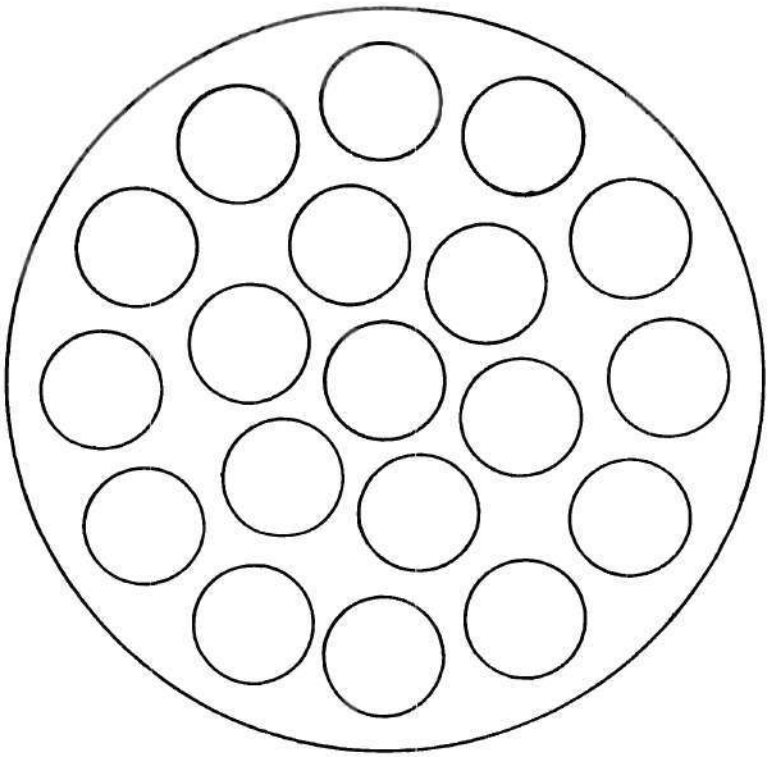
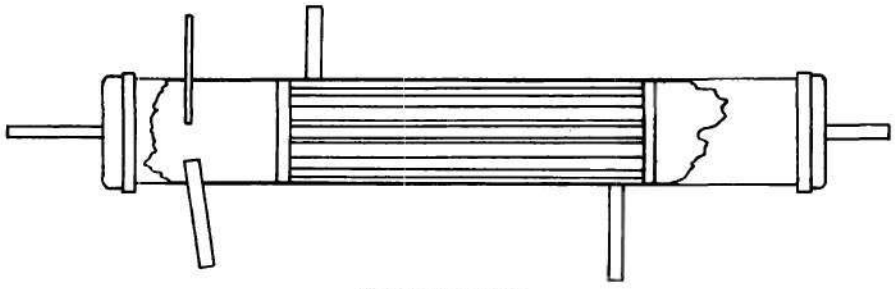



FIGURE 8
SLIDE VALVE



PLAN VIEW OF
TUBE SHEET

SCALE: FULL SIZE



CONDENSER
CUT-AWAY VIEW

SCALE: $1\frac{1}{2}$" = 1 FT.

FIGURE 9
LIQUID PRODUCTS CONDENSER

and fitted with standard pipe caps into which had been welded nozzles. The nozzle at the right, the entrance for overhead gases, was 3/8 inch and that at the left, the exit for condensed liquid, was 1/4 inch pipe. The 3/8 inch pipe welded into the disengaging section was for carrying condensed gases to the orifice meter. It was placed at a slight angle so that any entrained liquids might run back into the disengaging section. There was also a copper tubing welded into the disengaging section for a thermometer well. Two 1/2 inch nozzles were welded into the condenser shell, as shown, for the entrance and exit of the cooling water, flow being counter-current to the material on the hot side.

Figure 10 is a photograph of the unit's control board, manometer board, and other control facilities. The control board itself was a sheet of transite, which was supported by a simple wooden framework to which it was fastened by a series of screws along its edge. The multiple switch for selecting thermocouples to be read was mounted at the lower left-hand corner of the board. The two wires coming from one side of this switch were extended to the potentiometer. The potentiometer was a standard portable model, set on top of the control board and supported in position by the shorth length of angle iron mounted just above the switch. Just above the angle iron and to its right can be seen the dial and control handle of the autotransformer which supplied the current for the auxiliary heaters. At the right are the two duplicate sets of

control and indicating equipment for reactor heaters No. 1 and 2. Each set consists of a Variac autotransformer, an ammeter, a voltmeter, and a switch for the meter circuit. All of these units were mounted directly on the transite board. Centered above the two sets were the pilot light and the master switch for all three electric heater circuits. The pilot light was wired so as to indicate the position of the master switch.

The large piece of plywood mounted on the vertical member of the framework is the monometer board. The two double monometers at the top of the board were connected to the orifice meters; the left monometer connected to orifice meter No. 1 which measured the fluidizing gases as they entered the unit. The right monometer was connected to orifice meter No. 2 and was used to measure the residual gases leaving the unit. Each of these monometers consisted of two tubes so that it was possible to measure the gauge pressure at the down-stream orifice tap, as well as the differential pressure a-cross the orifice. The inclined monometer at the bottom of the monometer board was connected across the two ends of the reactor for measuring the pressure drop across the reactor. All of the monometer connections were made with copper tubing and the copper tubing was joined to the glass monometers by sections of rubber tubing.

The fluidizing gas for the destructive distillation runs was supplied from the cylinders of high pressure nitrogen

shown at the right. The pressure of the nitrogen coming from the cylinder was regulated by a standard pressure reducing valve. The gas from the reducing valve was piped to the manifold which was mounted on the back of the monometer board. The manifold was a piece of 1 1/4 inch pipe almost a foot long and threaded and capped at both ends. Along its length, it was drilled and tapped to receive two 1/4 inch and one 3/8 inch nipples. Valves were mounted on these nipples and are visible in the picture along the right edge of the monometer board. The two 1/4 inch valves control the flow of gas which served to keep the reactor pressure drop taps free from clogging by the sawdust. The lower 3/8 inch valve was for the purpose of controlling the flow of fluidizing gases. The 3/8 inch valve was connected to the vertical pipe which passes down behind the control board and is connected to orifice meter No. 1. Orifice meter No. 1 was mounted in a horizontal position under the monometer board behind the control stand. The outlet of orifice meter No. 1 was connected with 3/8 inch pipe directly to the expander in the bottom of the reactor.

Just to the left of the monometer board in the photograph, Figure 10, can be seen the liquid product collector. It is a glass vessel made from a section of about two inch tubing reduced at both ends to fit stopcocks. It was connected to the liquid products outlet of the condenser by a piece of rubber tubing and supported by two ordinary laboratory clamps which were attached to a rod. The rod was

Figure 10

Control and Measurement Facilities

welded to the condenser body.

Figure 11 is a front view of the whole apparatus except for part of the hopper-valve auxiliaries on top. This picture offers a good illustration of the method used in mounting the apparatus. The tips of the wings, which are welded to the disengaging section above the reactor, can be seen protruding from the insulation and resting on the top horizontal bars of the framework. The formed metal strap, which is clamped on the apparatus just below the bottom slide valve and attached to the bottom horizontal members of the framework, was placed there for the purpose of maintaining the alignment of the reactor and to hold it steady. The insulation covering on the reactor, the disengaging section, and their connecting reducer was glass wool in two layers having a total thickness of about three inches. The insulation on the cyclone and the pipe connecting it to the disengaging section is only a very heavy layer of asbestos paper formed to fit.

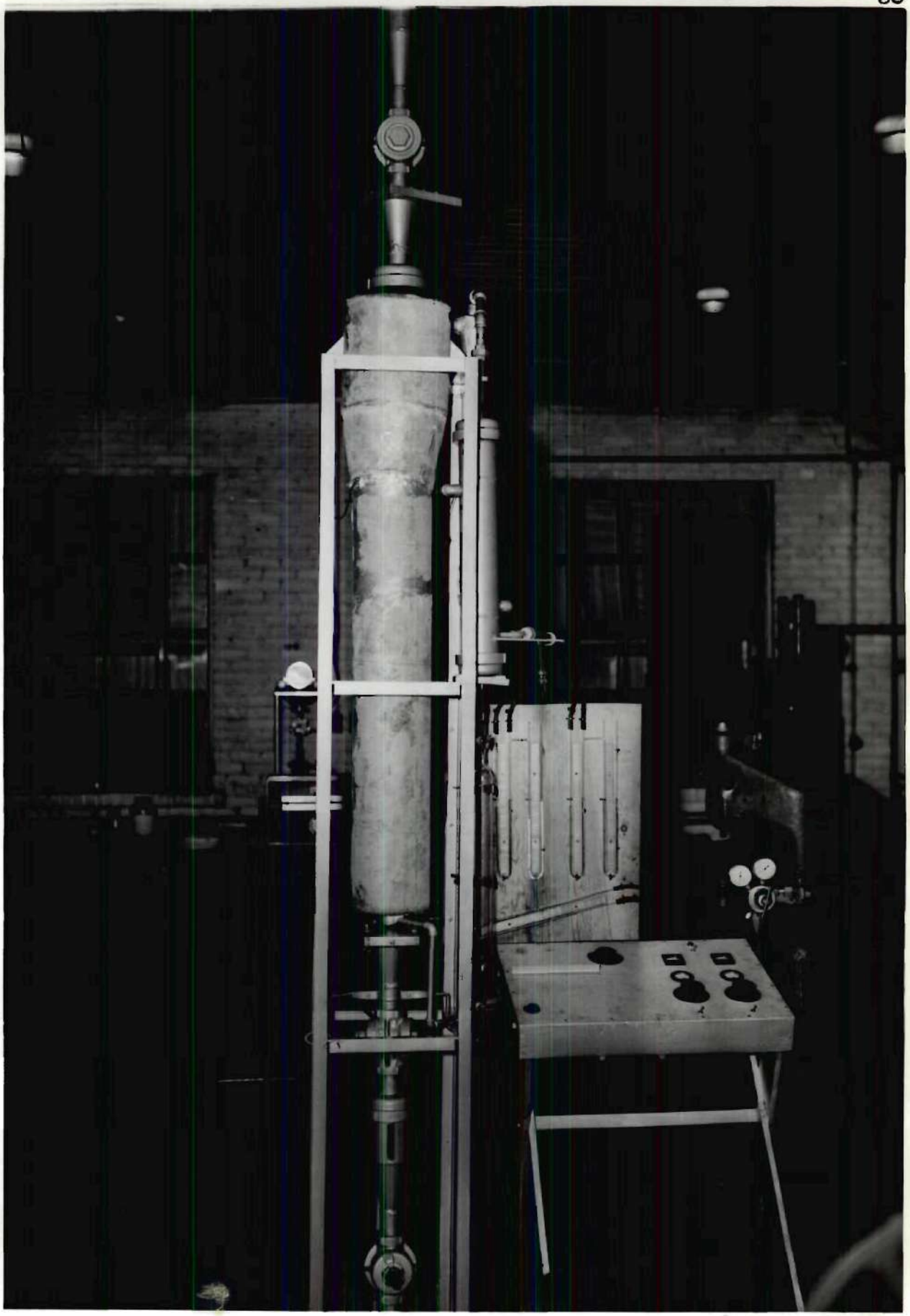


Figure 11
View of Apparatus Showing Mounting of Units on the Framework

# Chapter IV

## Operation and Results

In preparing for the operation of the apparatus, the first step necessary was the calibration of the orifice meters and the thermocouples. Since only an approximate calibration was desired at the time, it was not considered necessary to use the most accurate methods of calibration. The thermocouples were checked by being immersed in a beaker of water simultaneously with a thermometer. Checks were made at several temperatures with each thermocouple and the thermocouples were found to be correct to within about one degree centigrade. The two orifice meters were checked by piping each in series with a positive displacement gas meter obtained from the Atlanta Gas Light Company. A series of runs was made with each orifice meter, using nitrogen from a high pressure cylinder as the sourse of the gas. The data obtained are tabulated in the appendix with a plot of the volume figures obtained converted to velocities in the reactor at $27^{o}$ C.

The second step preparatory to operation was to prepare adequate quantities of suitable sawdust for the contemplated runs. While the apparatus was actually designed to study destructive distillation of soft woods such as yellow pine, for the preliminary runs it was decided that a hard wood should be used since it was contemplated that hard wood would be easier

to work with. A good sized piece of secondhand red oak was obtained from a lumber yard. No moisture determination was made in the wood or the sawdust therefrom; however, this wood had been used in a building for a number of years, so it was well dried. The piece of wood was turned down on a lathe to obtain a quantity of chips, and the chips were further ground to a powder in a rotating hammer mill. The powder, thus obtained, was screened in a series of screens of varying sizes producing a number of fractions, each of which covered a range of screen mesh sizes.

The first operation of the unit was with air as a fluidizing medium and sawdust of relatively large sizes. This operation was solely for the purpose of preliminary familiarization with the apparatus; no heat was used; and no data were taken. The sawdust used passed a 20 mesh, but was retained on 42 mesh screen. A little trouble was experienced in over loading the second feed hopper and the withdrawal hopper, in that they plugged up a bit, but, on the whole, this operation was quite satisfactory. A recovery of some small material was made by the cyclone and the overall recovery was good.

A second preliminary run was made on the unit using sawdust which passed a 42 mesh screen, but was retained on 100 mesh. The main purpose of this run was to check on the capacity of the reactor. Heat was also applied during this run to check operation of the thermocouples and to further

dry the insulation. Since the heating caused a certain amount of combustion, it was not possible to obtain any good data on the reactor capacity. It was discovered in this run, however, that the fluid in the reactor pressure drop manometer was not heavy enough, so, at the end of the run, the water in this manometer was removed and mercury substituted therefor. This run indicated satisfactory operation of all the other equipment.

The first run for actual destructive distillation was made using 42 to 100 mesh red oak sawdust and nitrogen. According to the literature (5) the temperature to be sought for destructive distillation was about 280°C. The object of this run was to try to attain reaction at this temperature. It was possible to reach this temperature and also to exceed it considerably, as indicated by the tabulated data in the appendix. It was not possible to condense an appreciable amount of liquid product. Apparently, however, a good amount of product was produced, for there was a production of copious quantities of noxious smoke from the outlet of orifice meter No. 2. The poor recovery also substantiates this idea. Of the 418 grams of sawdust charged, only 161 grams of solids and liquid were recovered. Of the recovery, 133 grams were carbonized sawdust which came out of the bottom of the reactor, and only 10 grams were liquid condensed in the receiver.

During this run it was noticed that the pipe from the nitrogen cylinder was so cold from the expanding gas that water

condensed on its surface. It was also noted that the action of the material in the fluidized bed caused rapid pulsations in the monometers of the orifice meters, and, when a slug of solids entered the reactor suddenly as in adding a large amount of charge, the magnitude of the pulsations might be so great as to blow the fluid out of the monometer. This was especially true in the gauge pressure half of the monometer for orifice meter No. 1; therefore, that tube was filled with ethylene dibromide at the end of this run. During the run, the water which had been in the monometer was blown out.

At the close of the run, it was noticed that the flow of gas through the apparatus was somehow blocked. When the union in the pipe connecting the disengaging section to the cyclone separator was broken, it was noted that this pipe was full of a substance which appeared to be a combination of fine sawdust and a tar-like material. After removing this accumulation, the cyclone and the liquid products condenser were inspected and found to be in good condition.

The lack of production of liquid product during this run was puzzling, since the thermometer in the bottom of the liquid products indicated temperatures varying only between about 25 and 27° C. This temperature range was almost identical with room temperatures at the time.

The second and last destructive distillation run was made using 42 to 100 mesh sawdust and nitrogen as the

fluidizing medium. It was decided, however, to make this run at lower temperatures and gas velocities, in order to try to get better control of the operation. During this run all the monometers were filled with colored water except the right half of the monometer of orifice meter No. 1, which contained ethylene dibromide, and the reactor pressure drop monometer which contained mercury.

During this run, there was much better agreement attained among the four thermocouples of the reactor, than during the previous run; however, none of them went above 200°C. A certain amount of smoke was produced during this run, but not nearly as much as in the previous run. A much better recovery was made on this run, since 89.9% of the 526 grams charged were accounted for as products. In contrast to the black, completely carbonized bottom product obtained in the previous run, the product from this run showed much the color of the original sawdust, although it definitely was partially carbonized. Again in this run, the recovery of liquid was poor, even though condenser outlet temperatures were down around 27°C; however, the recovery in the run was relatively much better than in the previous one. The liquid which was collected in both runs had a watery consistency and a brownish color.

The operation and control of the apparatus seemed to be quite satisfactory, although the experimental results obtained were not all that might be hoped for. During the last

run, it was possible to bring the reactor thermocouples within better agreement, and the solid product obtained indicated that control of the degree of destructive distillation could be obtained by controlling the temperature. The manner in which production of smoke and liquid product increased immediately after addition of fuel charge, indicated that the reactions taking place must be quite rapid. Perhaps this was more a matter of driving off the free water from the wood, rather than actual destructive distillation.

Each run of about two hours' duration consumed one cylinder of nitrogen. In starting each run, the nitrogen was run through the unit first at low velocities before the heat was turned on. After the unit had heated up a bit, the nitrogen velocity and the heat were adjusted. When the desired adjustment was attained, the first charge of sawdust was put in and the run was made. Further adjustments and additional charges were made during the run, as deemed necessary. At the end of the run, the gas velocity was reduced in order to remove all the solids remaining in the apparatus. The flow of gas was momentarily stopped to remove the last of this material, and then it was resumed when the heat was turned off. Gases were blown through the apparatus during the entire cooling period, in order to make the cooling more uniform and more rapid. All of the recovered products from each run were weighed to check the yield of the run.

# Chapter V

## Conclusions and Recommendations

The experimental runs made with this apparatus indicated that it is usable for its purpose, but that it could well be improved by some alterations. In particular, the reactor, the reacter expander, the disengaging section, the cyclone separator, and the measurement and control equipment seemed to be quite satisfactory; however, the hopper-valve combinations at the top and the bottom and the condenser did not seem to be completely satisfactory.

During batch runs, such as the two made, the operation of the hopper-valve combinations did not necessarily have to be smooth; however, for operating the apparatus in continuous runs, they would have to operate without plugging or unevenness in flow. The operation of these facilities during the charging and discharging of the unit showed that they were not operating as they should. It is believed that the installation of several taps, or entrances, at strategic points among the hoppers and valves for the application of aeration gases would promote better action. By means of this aeration, the material in the hoppers and valves would be fluidized. Although it would be only slightly fluidized, not nearly as well aerated as the material in the reactor,

its flow and the operation of the slide valves would be greatly facilitated.

The greatest deficiency noted in the destructive distillation runs made was the apparant poor recovery of liquid product, in spite of cooling to room temperature in the condenser. While this may have been only because of not knowing how to operate properly, it does seem that the trouble may be in either not having sufficient cooling for the operating pressure, or in having such a large proportion of nitrogen to the amount of liquid product present in the vapor. In order to arrive at a more definite answer to this question, it would be necessary to make a number of additional experimental runs. Two runs do not produce sufficient data to arrive at very definite conclusions. Additional runs would also make it possible to operate at a number of different conditions and note the effect of the various conditions on the yield of liquid product.

A construction and maintenance trouble which might also well be eliminated is the unwieldiness of attaching and removing the top hopper-valve facilities by means of the five inch pipe cap which threads onto the top of the disengaging section. If a regulation flange were screwed onto, or otherwise attached to the top of the disengaging section, and a properly drilled regulation blind flange were substituted for the pipe cap, the top hopper-valve auxiliaries could then be attached to the rest of the apparatus with comparative dispatch

and ease. Such a flanged arrangement with proper metal gasket would also simplify the problem of maintaining a gas-tight joint at this point.

# BIBLIOGRAPHY

(1) Aries, R. S., Polytechnic Institute of Brooklyn, before Northeastern Wood Utilization Council, Boston, December 12, 1946. Chem. Eng., 54, No. 1, 238 (1947).

(2) Blanding, F. H., and Roetheli, B. E., "Retorting Oil Shale by the Fluidized Solid Technique." Oil Gas Journal 45, No. 41, 84-8,96 (1947).

(3) Carlsmith, L. E., and Johnson, F. B., "Pilot Plant Development of Fluid Catalytic Cracking." Ind. Eng. Chem. 37, 451-5 (1945).

(4) Daniels, L. S., "Shell Incorporates Design Refinements in Fluid Catalytic Cracking Unit at Deer Park." Pet. Ref., 26, No. 2, 95 (1947).

(5) Hawley, L. F., "Wood Distillation." The Chemical Catalog Company, Inc., New York (1923).

(6) Kalbach, J. C., "Fluidization in Chemical Reactions." Chem. Eng. 54, No. 1, 105 (1947)

(7) _____, "Handling Solids - Gas Reaction by Fluidization". Chem. Eng. 54, No. 2, 136 (1947).

(8) MacDonald, M., "Automatic Control of Fluid Catalytic Cracking Unit." Pet. Ref. 25, No. 10, 87 (1946).

(9) Mateer, G. L. and Haney, J. D., "Three Fluid Catalytic Cracking Units Form Heart of New Refinery." Oil Gas Journal, 43, No.9, 87 (1944).

(10) Murphree, E. V. et al. "Fluid Catalyst Cracking for Premium Fuels." Oil Gas Journal, 42, No. 28, 37 (1943).

(11) _____, "Improved Fluid Process for Catalytic Cracking." Trans. A. I. Ch. E., 41, 19 (1945).

(12) Murphree, E. V. et al. "Improvements Increase Efficiency of Fluid Catalytic Cracking Process," Oil Gas Journal, 43, No. 43, 64-81 (1945).

(13) _____, "Fluid Catalyst Cracking for Premium Fuels." Pet. Ref. 22, 357-64 (1943).

(14) _____, "War Developments in the Petroleum Industry." Ind. Eng. Chem. 35, 623 (1943).

(15) _____, "Fluid Catalyst Process - Catalytic Cracking of Petroleum." Ind. Eng. Chem. 35, 768 (1943)

(16) Russell, R. P., "The Genesis of a Giant." Pet. Ref. 23, No. 1, 91 (1944).

(17) Thomas C. L., et al. "Cracking With Catalysts." Oil Gas Journal 42, No. 27, 131 (1943).

(18) _____, and Hoekstra, James, "Fluidized Fixed Bad Method for Contacting Solids with Gases and Vapors." Ind. Eng. Chem. 37, 332-4 (1945).

(19) Wickham, H. P.,"Mechanism of Flow in Fluid Catalyst Cracking." Pet. Ref. 24, No. 7, 107 (1945)

# A P P E N D I X

Data - Test run No. 1 of destructive distillation, May 21, 1947
Sawdust 42 - 100 mesh. Nitrogen - fluidization agent.

| I | II | III | IV | V | VI | VII A | VII B | VIII A | VIII B | IX | X | XI | XII | Amt. of Sawdust Added |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1405 | 73 | 158 | 120 | 107 | 84 | 1.9 | 7.3 | 1.6 | 0.6 | 0 | 30 | 30 | 97 | |
| 15 | 85 | 146 | 120 | 110 | 86 | 1.9 | 10.0 | 1.5 | 0.7 | 1.5 | 30 | 30 | 97 | 112 gm |
| 25 | 94 | 129 | 120 | 112 | 90 | 1.9 | 15 | 1.4 | 0.5 | 3.4 | 50 | 50 | 107 | 96 gm |
| 35 | 105 | 127 | 127 | 119 | 95 | 1.8 | 19 | 2.0 | 0.7 | 5.4 | 60 | 60 | 107 | 100 gm |
| 45 | 132 | 150 | 148 | 134 | 101 | 1.8 | 19 | 1.8 | 0.7 | 4.8 | 70 | 70 | 107 | |
| 48 | 148 | 176 | 166 | 152 | 110 | 1.8 | 19 | 1.8 | 0.7 | 4.8 | 80 | 80 | 112 | |
| 55 | 201 | 219 | 202 | 182 | 123 | 1.4 | 17 | 1.4 | 0.7 | 4.2 | 90 | 90 | 112 | |
| 1503 | 262 | 281 | 253 | 229 | 137 | 1.5 | 17 | 1.4 | 0.7 | 2.8 | 80 | 80 | 112 | |
| 10 | 288 | 309 | 279 | 256 | 147 | 1.4 | 16 | 1.6 | 0.6 | 2.6 | 75 | 75 | 112 | |
| 25 | 280 | 334 | 308 | 298 | 168 | 1.4 | | 1.6 | 0.6 | 3.2 | 75 | 75 | 112 | |
| 36 | 323 | 356 | 319 | 307 | 189 | | | 1.0 | 0.4 | 4.1 | 70 | 70 | 112 | 109 gm |
| 50 | 292 | 324 | 300 | 294 | 190 | | | 1.1 | | 7.8 | 0 | 0 | 0 | |
| 1604 | 286 | 319 | 292 | 285 | 191 | | | 1.0 | 0.4 | | 70 | 70 | 112 | |
| 10 | 303 | 338 | 301 | 295 | 195 | | | 1.0 | 0.4 | | 70 | 70 | 112 | |

Total Charge 417 gm
Liquid Product 10 gm
Solids, Product 133 gm
Solids (Cyclone) 18 gm
Total Recovered 161 gm

Column

I - Time - hours
II - Temperature, °C, bottom of reactor
III - " " middle of reactor
IV - " " next to top of reactor
V - " " top of reactor
VI - " " in center of disengaging section
VII A - Head, $cmH_2O$, orifice meter No. 1 differential pressure
VII B - " " " " " down stream tap, guage pressure
VIII A - " " " " No. 2 differential pressure
VIII B - " " " " " down stream tap, guage pressure
IX - Head, $cmH_2O$, differential pressure across reactor
X - Voltage across Heater No. 1
XI - " " " No. 2
XII - " " Auxiliary Heater

Data - Test run No. 2 of destructive distillation, May 22, 1947
Sawdust 42 - 100 mesh. Nitrogen - fluidization agent.

| I | II | III | IV | V | VI | VII A | VII B | VIII A | VIII B | IX | X | XI | XII | Amt. of Sawdust Added |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1533 | 96 | 198 | 138 | 116 | 74 | 0.9 | 3.7 | 0.8 | 0.2 | 0 | 40 | 40 | 112 | |
| 44 | 108 | 182 | 147 | 127 | 81 | 1.1 | 14 | 1.3 | 0.3 | 6.5 | 40 | 40 | 112 | 210 gm |
| 49 | 135 | 174 | 151 | 134 | 88 | | | | | | | | | |
| 51 | 139 | 171 | 158 | 139 | 93 | | | | | | | | | |
| 59 | 142 | 170 | 160 | 144 | 97 | 1.2 | | 1.1 | 0.3 | 6.2 | 45 | 45 | 112 | |
| 1607 | 138 | 156 | 152 | 146 | 105 | 1.3 | 22 | 1.4 | 0.5 | 14.5 | 45 | 45 | 112 | 204 gm |
| 16 | 149 | 162 | 160 | 155 | 114 | 1.3 | 22 | 1.2 | 0.4 | 15.0 | 50 | 45 | 112 | |
| 26 | 157 | 170 | 167 | 160 | 122 | 1.2 | | 1.3 | 0.4 | 10.8 | 50 | 45 | 112 | |
| 45 | 178 | 190 | 188 | 180 | 132 | 1.2 | 24 | 1.3 | 0.4 | 12.2 | 45 | 45 | 112 | |
| 52 | 181 | 192 | 192 | 182 | 136 | | | | | | | | | |
| 1700 | 185 | 199 | 193 | 182 | 140 | 1.0 | 24 | 1.0 | 0.4 | 11.3 | 40 | 45 | 112 | |
| 15 | 179 | 190 | 187 | 180 | 146 | 0.8 | 32 | 1.0 | 0.4 | 14.5 | 45 | 45 | 112 | 112 gm |
| 23 | 181 | 191 | 191 | 186 | 150 | | | | | | | | | |
| 30 | 186 | 199 | 195 | 189 | 156 | 0.8 | 32 | 1.0 | 0.4 | 13.6 | 45 | 45 | 112 | |
| 36 | 188 | 201 | 198 | 192 | 156 | 0.8 | 32 | 1.0 | 0.4 | 13.6 | 45 | 45 | 112 | |
| 38 | 188 | 200 | 199 | 193 | 157 | 0.8 | 32 | 1.0 | 0.4 | 13.6 | off | off | off | |

| | |
|---|---|
| Total Charge | 526 gm |
| Liquid Product | 9 gm |
| Solids, Product | 445 gm |
| Solids, (Cyclone) | 20 gm |
| Total Recovered | 474 gm |

NB: For meaning of Roman numerals heading columns, see preceding page.

Data - Orifice meter calibrations using positive displacement meter from Atlanta Gas Light Company in series with orifice meter. See data plotted on following graphs.

| Orifice Meter No. 1 | | | | | Orifice Meter No. 2 | | | | |
|---|---|---|---|---|---|---|---|---|---|
| $I_A$ | $I_B$ | II | III | IV | $I_A$ | $I_B$ | II | III | IV |
| 1.1 | 1.8 | 19.3 | 0.5 | 1.112 | 0.8 | 0.6 | 22.8 | 0.5 | 0.941 |
| 0.6 | 0.8 | 33.1 | 0.5 | 0.648 | 0.3 | 0.2 | 37.2 | 0.5 | 0.577 |
| 6.8 | 10.0 | 8.7 | 0.5 | 2.47 | 1.6 | 0.6 | 18.2 | 0.5 | 1.179 |
| 3.8 | 5.3 | 12.5 | 0.5 | 1.717 | 2.9 | 1.0 | 14.4 | 0.5 | 1.490 |
| 10.2 | 14.8 | 32.2 | 2.0 | 2.66 | 3.9 | 1.4 | 12.6 | 0.5 | 1.703 |
| 3.0 | 4.3 | 14.5 | 0.5 | 1.481 | 6.0 | 2.2 | 10.1 | 0.5 | 2.124 |
| 1.5 | 2.0 | 20.5 | 0.5 | 1.047 | | | | | |
| 4.4 | 6.4 | 23.2 | 1.0 | 1.848 | | | | | |
| 9.5 | 14.4 | 8.2 | 0.5 | 2.62 | | | | | |
| 7.0 | 10.2 | 9.0 | 0.5 | 2.38 | | | | | |
| 3.0 | 4.5 | 13.5 | 0.5 | 1.590 | | | | | |
| 1.4 | 2.0 | 21.2 | 0.5 | 1.012 | | | | | |
| 0.4 | 0.6 | 41.2 | 0.5 | 0.521 | | | | | |

$I_A$ - Differential pressure across orifice, $cmH_2O$

$I_B$ - Downstream gauge pressure " "

II - Time for passage of measured amount of gas, seconds

III - Volume of gas during measured time, $(feet)^3$

IV - Velocity of gas through reactor at 27°C, feet per sec.

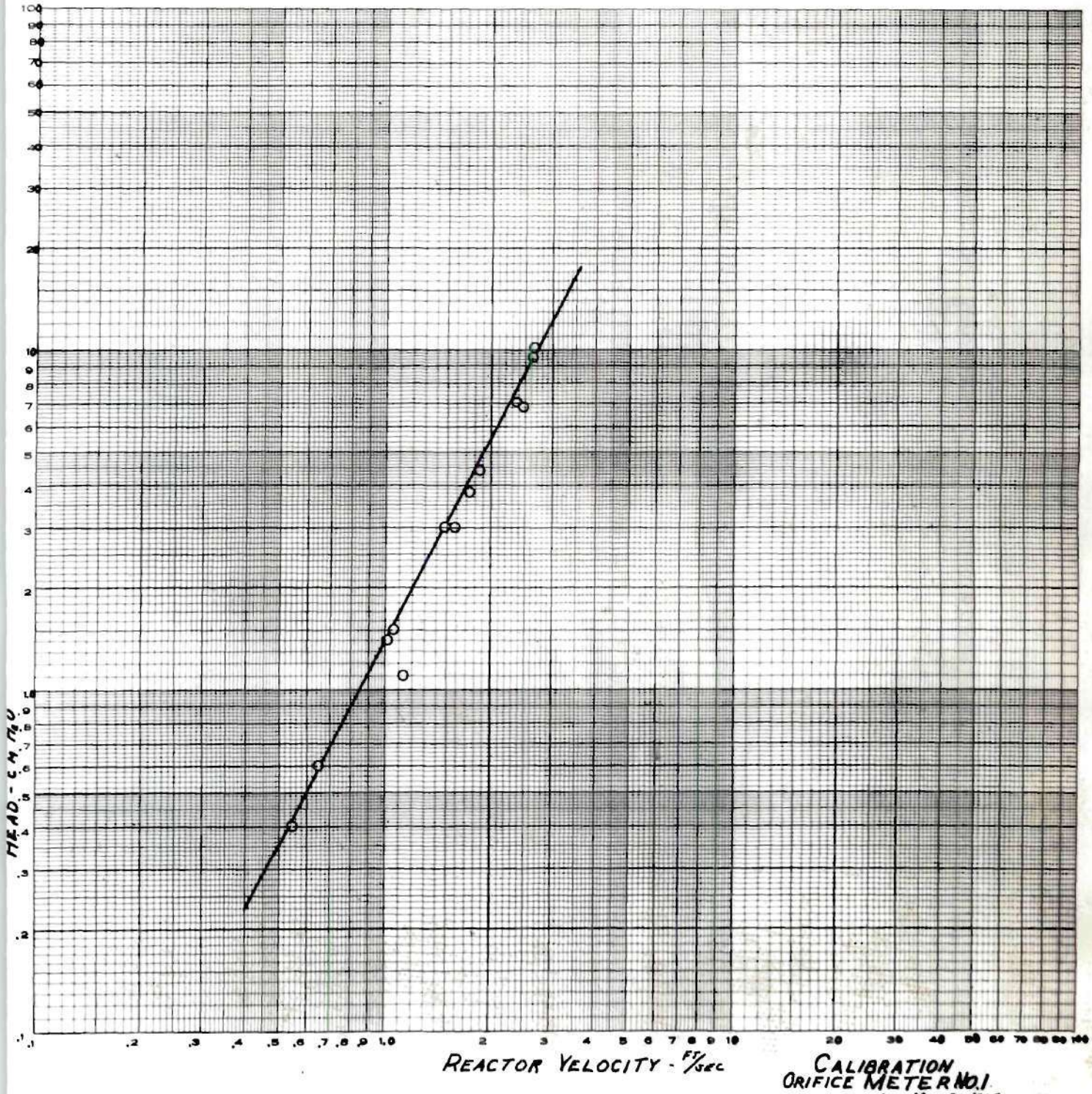

HEAD - C.M. Hg.
REACTOR VELOCITY - FT/SEC
CALIBRATION
ORIFICE METER NO. 1
R.P Jongedyk May 20, 1947

Figure 12

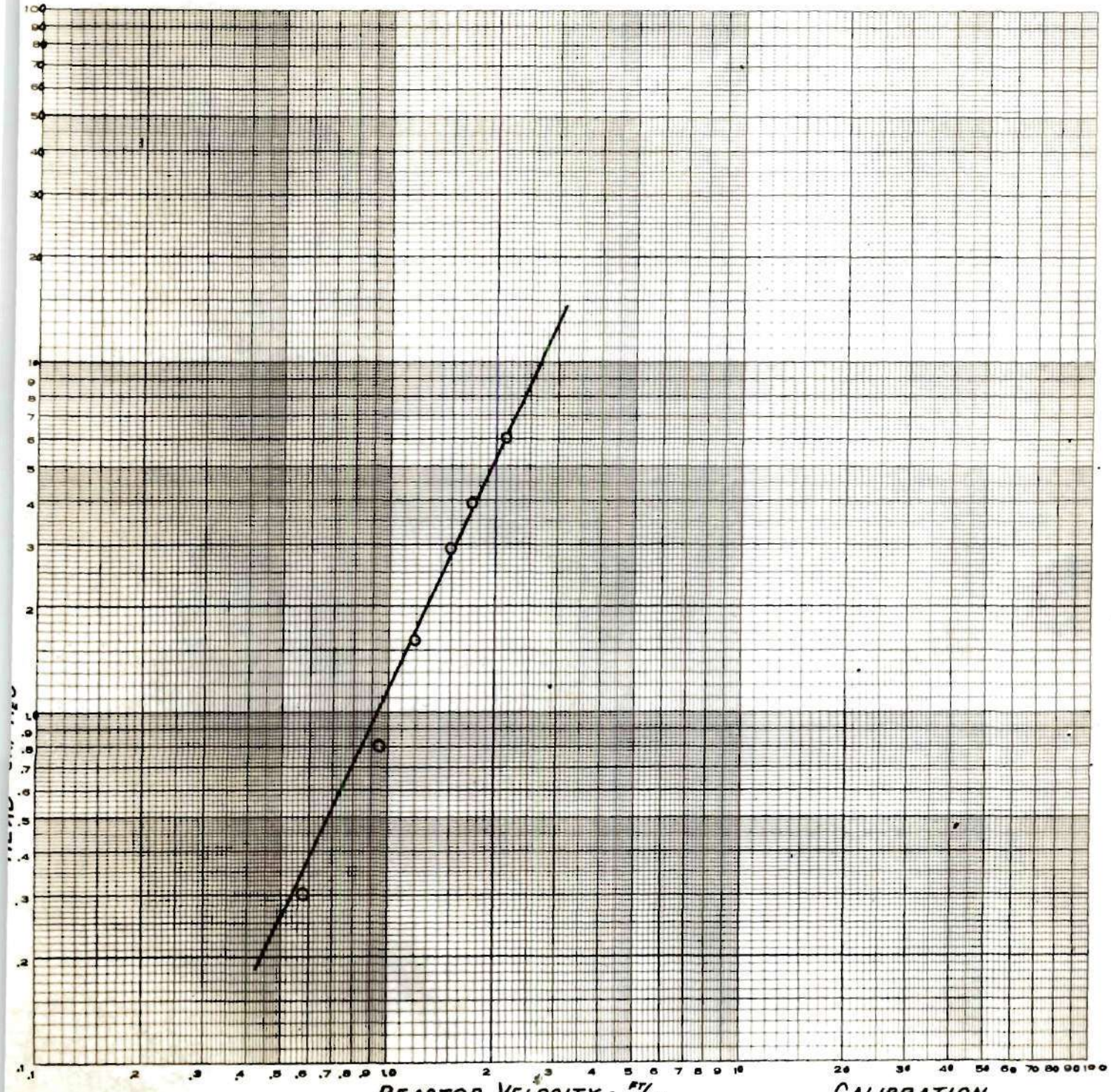

REACTOR VELOCITY - FT/SEC
CALIBRATION
ORIFICE METER NO. 2
R. P. Longwell May 20, 1947

Figure 13

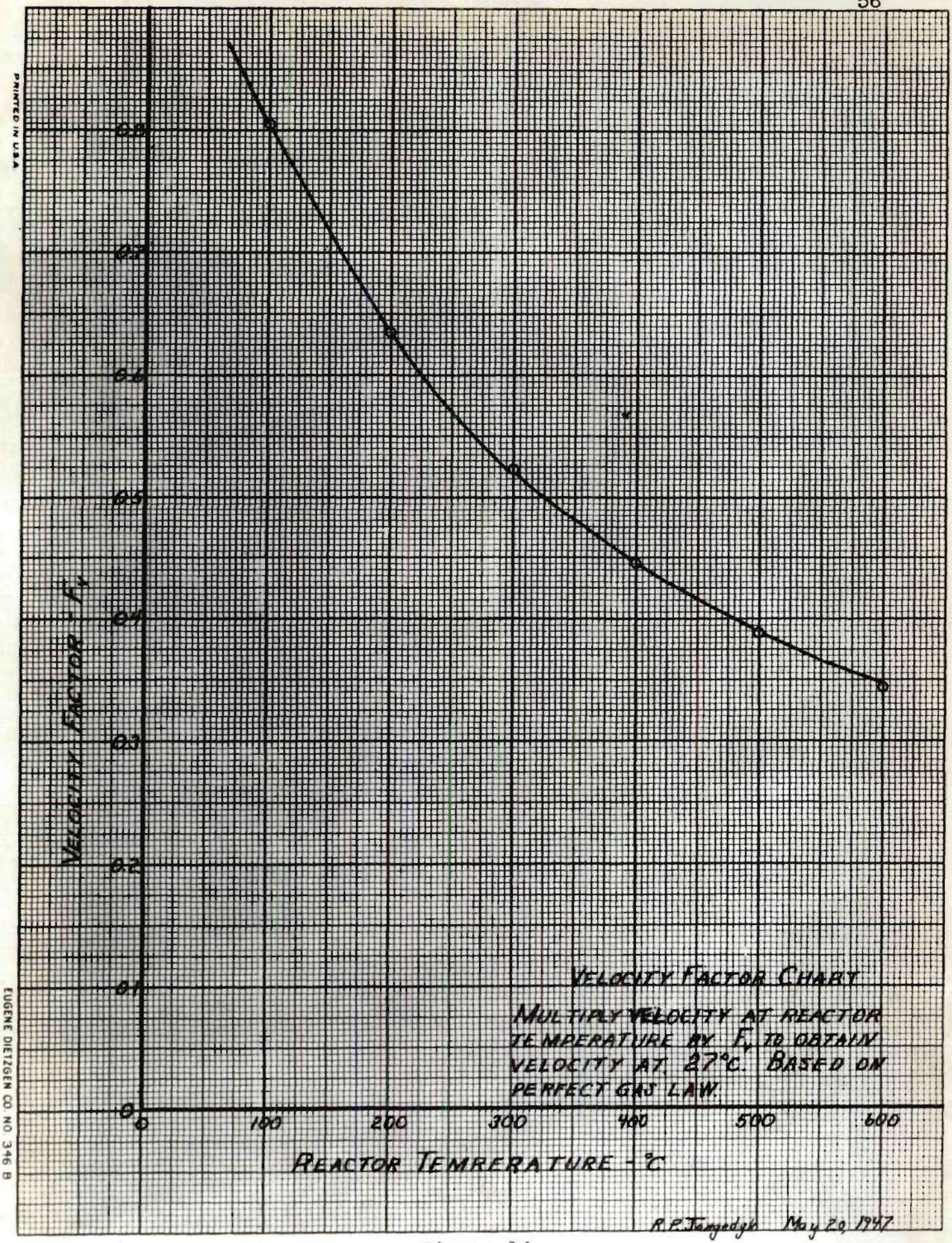



Figure 14